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TECHNICAL PROGRESS REPORT

Integrated Oxygen Recovery System

Technical Progress Report No. 2

Prepared Under

Program No. 1650

for

Contract NAS8-39843

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		LIST OF ACRONYMS				
	IORS In OGA C PEEK P SBIR S	Carbon Dioxide Reduction Assembly Integrated Oxygen Recovery System Oxygen Generation Assembly Olyetheretherkeytone Integrated Oxygen Recovery System Integrated Oxygen Recovery				

#### 1.0 WORK PERFORMED DURING REPORTING PERIOD

This Technical Progress Report summarizes the work performed under Contract No. NAS8-39843 from 03/09/93 through 05/07/93. This contract is a Phase I Small Business Innovation Research (SBIR) program to demonstrate the feasibility of the Integrated Oxygen Recovery System (IORS). The IORS is applicable to advanced mission air revitalization. It provides the capability for electrochemically generating metabolic oxygen  $(O_2)$  and recovering  $O_2$  from the space habitat atmosphere via a carbon dioxide  $(CO_2)$  reduction process within a single assembly. To achieve this capability, the IORS utilizes a novel Solid Metal Cathode (SMC) water electrolysis unit that simultaneously serves as the Sabatier  $CO_2$  reduction reactor.

The IORS would enable two major life support systems currently baselined in closed loop air revitalization systems to be combined into one smaller, less complex system. It would reduce fluidic and electrical interface requirements and eliminate a hydrogen ( $H_2$ ) interface. Furthermore, since the IORS utilizes an SMC, the system has the additional capability to generate high pressure  $O_2$  (i.e.,  $\approx 1,000$  psia) for recharging extravehicular activity  $O_2$  bottles. This capability is not part of currently baselined or planned technologies.

During this Phase I SBIR program we will evaluate the IORS process by demonstrating its performance and quantifying key system physical characteristics, including power, weight and volume.

Work performed during this reporting period included completion of the assembly of the Breadboard IORS, checkout and shakedown testing of it and its test setup, and the initial parametric testing.

#### 1.1 Assembly of the Breadboard IORS

Upon receipt of the polyetheretherkeytone (PEEK) to be used to construct the cap of the Breadboard IORS (Figure 1-1), the cap was fabricated. The Breadboard IORS was then assembled as described in the previous Bimonthly Technical Progress Report (TR-1619-1-1). It was integrated in the test setup (Figure 1-2, with legend in Figure 1-3).

A reference electrode, not shown in Figure 1-1, was fabricated and mounted in the Breadboard IORS. The electrode is a palladium/silver (Pd/Ag) tube, sealed at one end, and it has a geometric surface area of 10.1 cm<sup>2</sup>. It is inserted in the electrolyte adjacent to the anode. Prior to operation of the Breadboard IORS, the reference electrode is charged with hydrogen by applying a cathodic current of 0.87 A for 12 minutes. Thereafter, no current is applied to the reference electrode, and it provides a virtually stable reference potential that can be used to measure changes in the potential of the anode or cathode.

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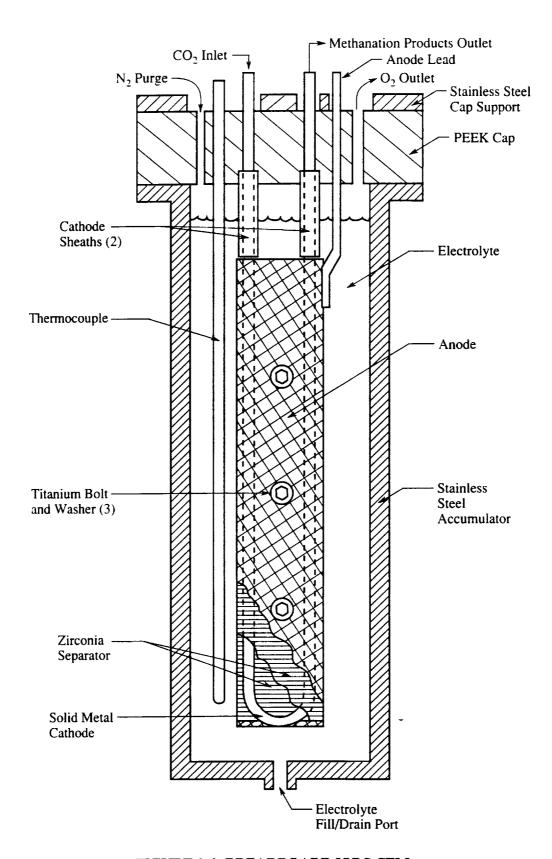


FIGURE 1-1 BREADBOARD IORS CELL

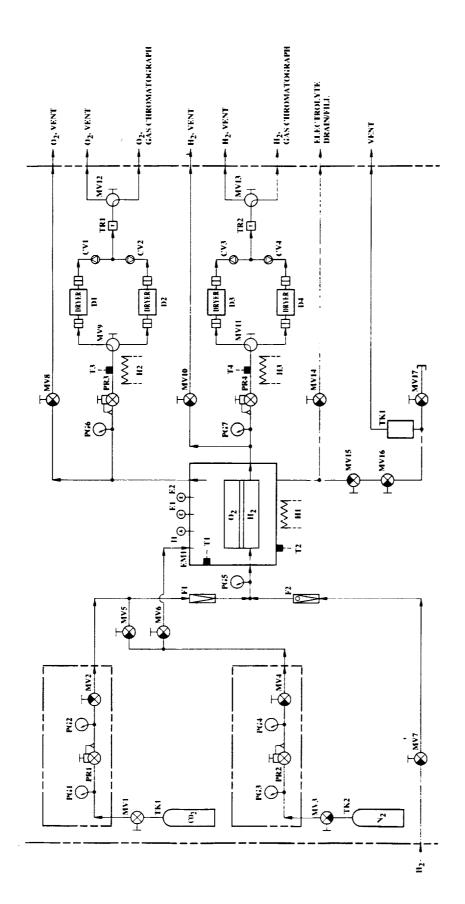


FIGURE 1-2 BREADBOARD IORS TEST SETUP MECHANICAL SCHEMATIC

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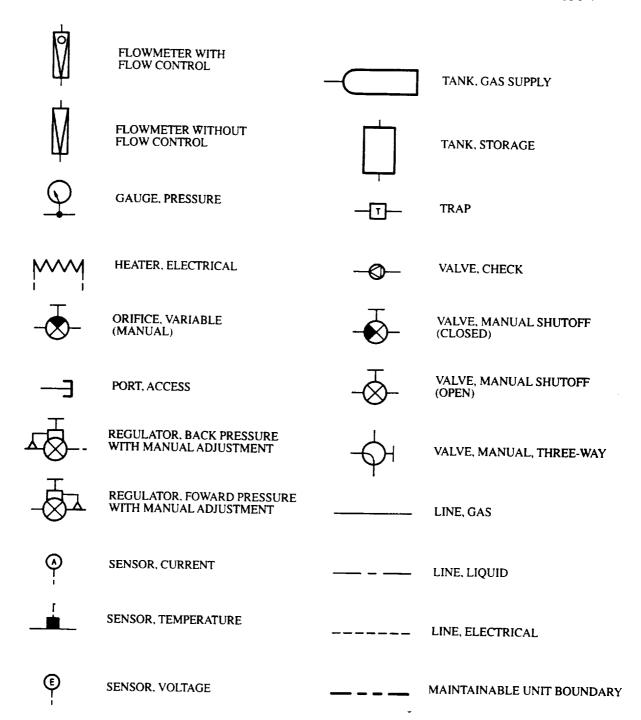


FIGURE 1-3 BREADBOARD IORS TEST SETUP MECHANICAL SCHEMATIC (LEGEND)

The gas chromatograph to be used for analysis of the product gases was also setup and calibrated during this reporting period.

### 1.2 <u>Checkout and Shakedown Testing</u>

These tests were performed to verify the integrity of the Breadboard IORS and its test setup. The tests were performed by operating the Breadboard IORS as an electrolyzer, without the methanation catalyst in the cathode.

During these tests it was found that the stainless steel accumulator was oxidized at 140 C, where it contacted the electrolyte (55% potassium hydroxide (KOH)). To avoid oxidation products from contaminating the electrodes, a Teflon cup was fabricated and inserted in the accumulator to hold the electrolyte.

During these tests it was also determined that the titanium nuts, bolts and washers (Figure 1-1) used to hold the anode against the separator and cathode also oxidized. These items were removed and replaced with nickel wire retainers that performed the same function.

After these changes, a new anode, cathode and separator were fabricated and mounted in the Breadboard IORS. No further oxidation products were detected in the electrolyte or on the electrodes. However, the zirconia cloth used as the separator tended to be torn where the anode and cathode were compressed against it. This occurred after six to ten hours of testing, and is attributed to the relatively low mechanical strength of the zirconia cloth when it is wet, compounded by the fact that the cathode expands and contracts as it absorbs and desorbs hydrogen.

The zirconia cloth was replaced with zirconia felt (0.127 cm thick). Further, the felt separator is replaced each time the Breadboard IORS is disassembled for electrolyte replenishment.

The electrochemical performance of the Breadboard IORS at 140 C is shown in Figure 1-4. The terminal voltage and the cathode-to-reference electrode potential is shown as a function of current density over the range of 45 to 200 mA/cm<sup>2</sup>. These data show that the cell potential is relatively low, and that there is little increase in the potential of the cathode, even at the relatively high current density of 200 mA/cm<sup>2</sup>.

Figure 1-5 shows the performance of the SMC in transferring  $H_2$  from its exterior to the interior of the SMC. The Breadboard IORS can produce  $H_2$  at a flow rate of more than 51 scc/min, with a transfer efficiency of 96% or greater, and a power consumption of only 13.5 W at 140 C.

Methanation of CO<sub>2</sub> in the SMC does not occur in the absence of catalyst. Prior to installation of catalyst in the Breadboard IORS, it was operated at a current density of 200 mA/cm<sup>2</sup> at a temperature of 143 C, with CO<sub>2</sub> flowing through the SMC at

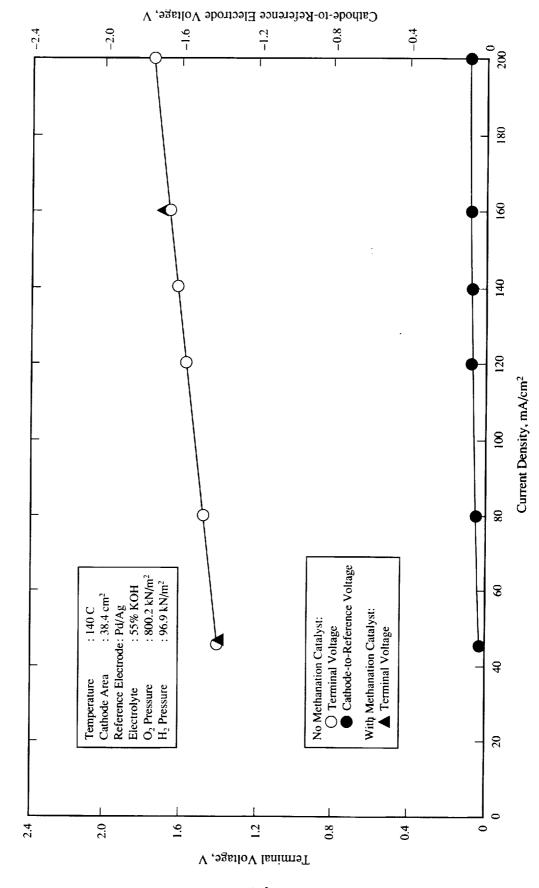


FIGURE 1-4 VOLTAGE AS A FUNCTION OF CURRENT DENSITY

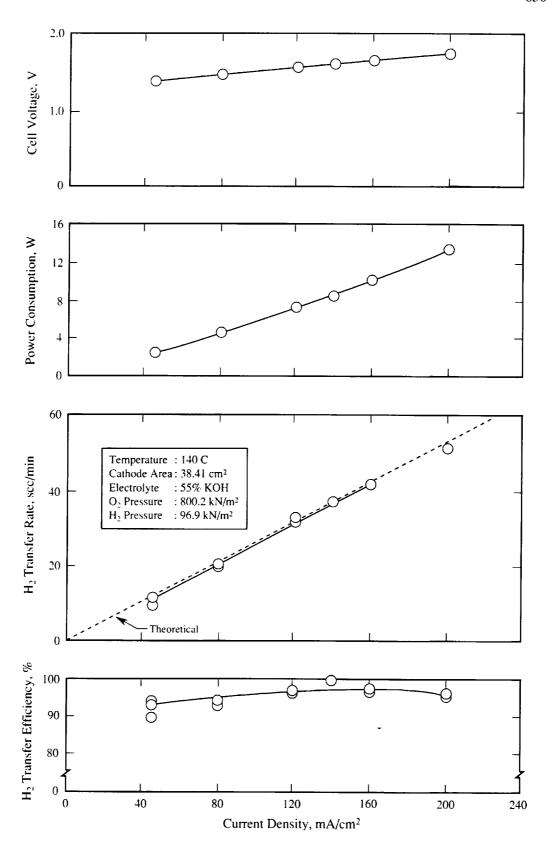


FIGURE 1-5 HYDROGEN TRANSFER CHARACTERIZATION

18.8 scc/min. This results in a molar ratio of  $H_2/CO_2$  of 2.5:1. No methane was detected by chromatographic analyses of the product gases.

Prior to the start of the parametric testing, the cathode was filled with the methanation catalyst. This catalyst is 20% ruthenium (Ru) on alumina extrudates (0.16 cm in diameter). The inner diameter of the SMC is 0.27 cm, so the catalyst extrudates had to be inserted coaxially in the SMC. The total weight of catalyst used in the Breadboard IORS is 1.47 g, and the length of the catalyst is 38.5 cm. The catalyst is retained in the cathode by plugs of Pyrex glass wool.

The presence of catalyst in the SMC does not affect its performance during the electrolyses of water, as shown by the two data points in Figure 1-4.

#### 1.3 <u>Parametric Testing</u>

The parametric testing involves studies of the effects of temperature, reactant gas composition and flow rate on the performance of the Breadboard IORS.

### 1.3.1 <u>Temperature</u>

Figure 1-6 shows the effects of temperature changes on the terminal voltage of the Breadboard IORS during the electrolysis of water. These data were obtained without CO<sub>2</sub> flowing through the cathode. Data are provided for two current densities, 45.6 and 160 mA/cm<sup>2</sup>. The decrease in terminal voltage with increasing temperature is -2.3 mV/C and -3.7 mV/C, respectively, for the two current densities.

The effect of temperature changes on the methanation reaction was studied through testing of the Breadboard IORS and use of the mathematical model for the IORS. The Breadboard IORS was operated at the conditions listed in Table 1-1, and the conversion efficiencies of H<sub>2</sub> and CO<sub>2</sub> to CH<sub>4</sub> were measured. The measured conversion efficiencies are compared in Figure 1-7 to the efficiencies calculated using the model. This figure shows excellent correlation of the performance predicted by the model and the actual performance of the Breadboard IORS over the range of operating parameters tested.

The effect of temperature on the performance of the Breadboard IORS, as predicted by the model, is presented in Figure 1-8. Curves are provided for inlet CO<sub>2</sub> flow rates of from 2 to 20 scc/min.

## 1.3.2 Reactant Gas Composition

The reduction of  $CO_2$  with  $H_2$  consumes four moles of  $H_2$  for every mole of  $CO_2$  that reacts. However, future space craft are expected to have less  $H_2$  available, so the methanation reaction will occur with a  $H_2/CO_2$  molar ratio of less than 4.0.

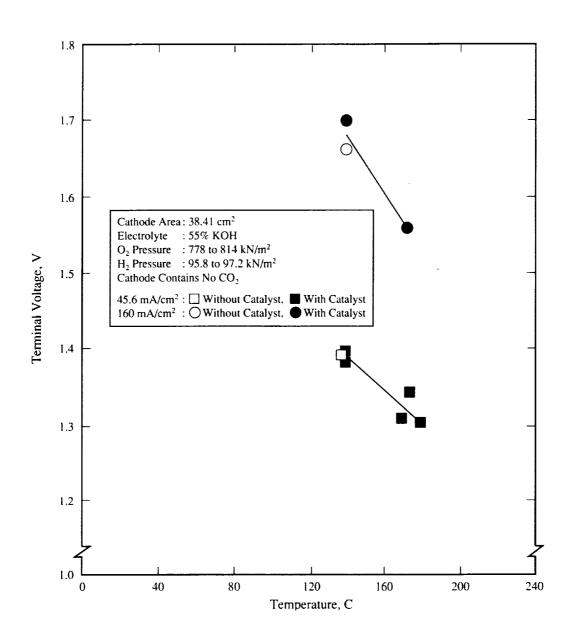


FIGURE 1-6 EFFECT OF TEMPERATURE ON ELECTROLYSIS VOLTAGE

TABLE 1-1 DATA FOR COMPARISON OF MEASURED AND CALCULATED CONVERSION EFFICIENCIES

a) Dioxide	alculated	5.81	6.16	13.0	17.2	66.5	4.60	22.7	100	28.5	24.6	34.9	34.3	37.5	36.3	85.4	48.6	69.4	81.6	49.2	50.4	6.76	53.3
ficiency, %(	Actual	5.33	5.48	10.4	11.8	46.8	4.23	27.4	100	34.2	29.4	36.1	34.9	36.5	35.9	80.4	51.7	73.6	83.1	49.8	51.8	96.4	29.0
Conversion Efficiency, % <sup>(a)</sup> drogen Carbon D	Calculated	11.2	11.7	13.4	15.7	13.3	11.6	26.7	47.6	60.4	62.7	46.8	44.6	41.7	41.6	39.7	67.5	64.0	62.0	63.3	63.0	63.3	62.2
O Hyd	Actual	10.4	10.4	10.7	10.8	9.4	10.6	32.0	47.5	72.3	74.7	48.4	45.3	40.6	41.1	37.4	71.7	8.79	63.1	63.9	64.5	62.2	8.89
	Temp., C	141.1	142.8	142.8	144.4	144.4	145.0	172.2	176.1	177.2	177.2	177.2	177.2	177.8	177.8	177.8	178.3	178.9	178.9	178.9	178.3	178.9	185.0
H <sub>2</sub> /CO,	Molar Ratio	2.05	2.11	3.88	4.37	20.0	1.59	3.40	8.41	1.89	1.57	2.98	3.08	3.60	3.49	8.60	2.88	4.34	5.26	3.11	3.20	6.19	3.43
· Initial CO,	Flow Rate, scc/min	19.1	18.6	10.1	7.96	2.03	24.0	10.5	1.14	5.95	6.26	6.47	9.76	6.61	08.9	2.90	3.47	2.69	2.32	3.80	3.70	1.71	3.89

(a) Catalyst = 0.76 cc of 20% Ru on Alumina. O<sub>2</sub> Pressure = 723 to 800 kN/m<sup>2</sup>. H<sub>2</sub> Pressure = 96 to 104 kN/m<sup>2</sup>. Electrolyte = 55% KOH.

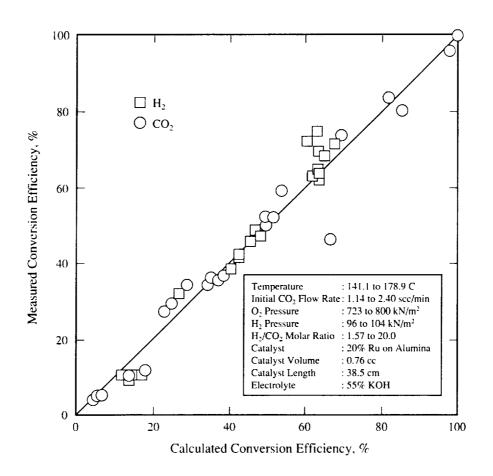


FIGURE 1-7 COMPARISON OF MEASURED AND CALCULATED CONVERSION EFFICIENCIES

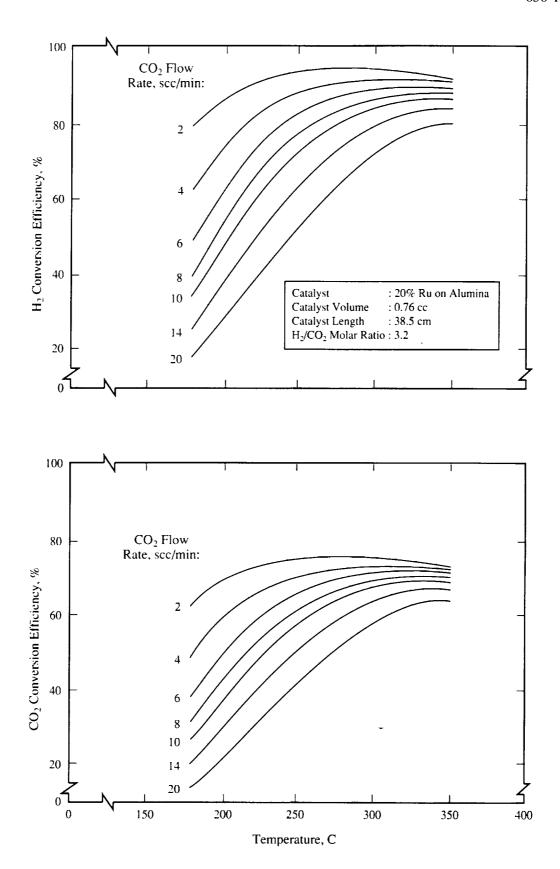


FIGURE 1-8 EFFECT OF TEMPERATURE ON METHANATION EFFICIENCY

To identify the molar ratio that may be expected in an end-item application, the specifications of Oxygen Generation Assembly (OGA) and the Carbon Dioxide Reduction Assembly (CReA) being developed for Space Station Freedom were reviewed. The OGA is to provide 4.65 kg O<sub>2</sub> per day during normal operation with a 4-person crew. The CReA is to process 4.0 kg CO<sub>2</sub> per day under normal (4-person) operating conditions. The CReA is to achieve 99% conversion of the lean feed constituent (i.e., H<sub>2</sub>). Both assemblies operate continuously.

With this information, the molar ratio anticipated in an end-item IORS, operating with a 4-person crew, was calculated to be 3.2:1, assuming 100% transfer efficiency of the  $H_2$  through the cathode.

Figure 1-9 shows the effect of the molar ratio on the conversion efficiency of the Breadboard IORS operating at 178 C. These data show the conversion efficiencies for both H<sub>2</sub> and CO<sub>2</sub>. Data are shown for values of the initial flow rate of CO<sub>2</sub> into the Breadboard IORS from 2 to 20 scc/min.

#### 1.3.3 Reactant Gas Flow Rate

As shown in Figure 1-8 there is a relationship between the flow rate of the gases and the efficiency of the conversion of the reactant gases to  $CH_4$  and  $H_2O$ . This is also shown in Figure 1-10 where the conversion rates of  $CO_2$  and  $H_2$  are shown as functions of the inlet flow rate of  $CO_2$ .

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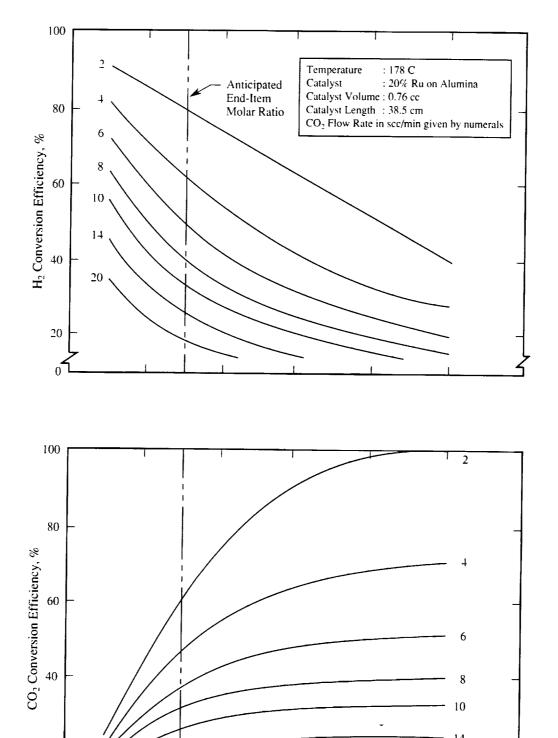


FIGURE 1-9 EFFECT OF REACTANT GAS COMPOSITION ON METHANATION EFFICIENCY

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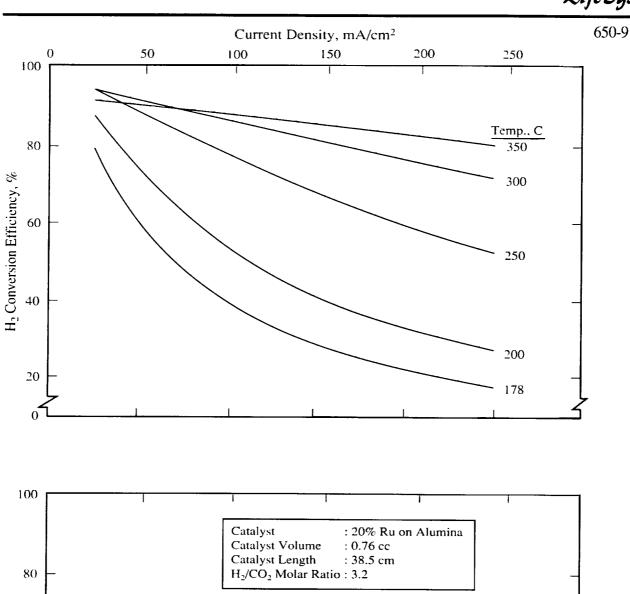
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H<sub>2</sub>/CO<sub>2</sub> Molar Ratio

8

20

10



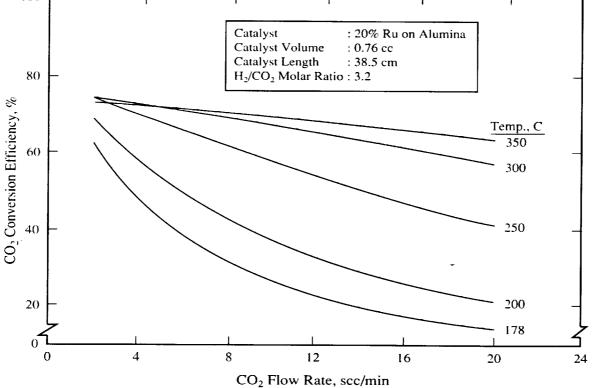


FIGURE 1-10 EFFECT OF INLET CO<sub>2</sub> FLOW RATE ON METHANATION EFFICIENCY

04/05/93

# 2.0 PROBLEMS

No problems have been encountered which may impede performance or impact program schedule or cost.

#### 3.0 WORK TO BE PERFORMED NEXT REPORTING PERIOD

The parametric tests will be completed during the next reporting period. Based on the analysis of those results and the results summarized here, the preliminary process design of a 4-person IORS will be developed. The preliminary mathematical models presented in the prior Bimonthly Technical Progress Report (TR-1619-1-1) will be updated if necessary. Also, the Final Report will be prepared and submitted on or before 07/08/93.

# 4.0 COST STATUS

The status of the program's cost is summarized below, as required by Attachment J-2 of Contract No. NAS8-39843.

1.	Total cumulative costs as of 04/30/93:	\$35,200
2.	Estimated cost to complete contract:	\$14,800
3.	Estimated percentage of work completed:	70.4%

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revitalization. The IORS provides habitat atmosphere via a carbon di a novel Solid Metal Cathode (SMC IORS enables two major life suppo	innovative Integrated Oxygen Reco the capability to electrochemically oxide (CO <sub>2</sub> ) reduction process within any water electrolysis unit that simultant out systems currently baselined in clo concept reduces fluidic and electric	generate metabolic oxygen (O <sub>2</sub> in a single assembly. To achievaneously serves as the Sabatier used loop air revitalization systems	) and recover O <sub>2</sub> from the space ve this capability, the IORS utilizes CO <sub>2</sub> reduction reactor. The terms to be combined into one
	uation of the IORS process directed ght and volume. In this report, the		
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